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John J. Scialdone Goddard Space Flight Center Greenbelt, Maryland



National Aeronautics and Space Administration

Goddard Space Flight Center Greenbelt, Maryland 20771



ABSTRACT

The sources of molecular deposits on a Temperature-Controlled Quartz Crystal Microbalance (TQCM) attached to the shuttle bay wall at some distance from the payload and pointing out of the bay have been investigated. Explored as possible sources are: (1) the outgassing of the shuttle tiles treated with the water proofing di-methyl-ethoxy-silane (DMES) compound, scattered back by the ambient molecules into the bay; (2) The ambient scattered return flux of the outgassing from the RTV 560 silicone which is used as the adhesive for the tiles; (3) the ambient scattered return flux of the payload outgassing; (4) the return flux from outgassing source originating near, below, or adjacent to the package containing the TQCM; and (5) the self-scattering of venting plumes and outgassing above the monitor package depositing on the TQCM. In all of these sources, the magnitudes of the outgassing, of the leakage rates, and venting had to be estimated in the absence of data. The resulting analyses which can be useful for similar investigations, indicates that the outgassing from the tiles, the RTV, the payloads, or the shuttle may not have been the sources of the deposit (about 1 μ g/cm² during the period from bay door opening and payload release from the bay). It appears more probable that the origin is the self- and/or ambient-scattered return fluxes originating near, below, or internal to the monitor package.

Introduction

The Temperature Controlled Quartz Crystal Microbalances (TQCM) sensors of the Interim Operational Contamination Monitor (IOCM) package installed in the bay of the STS-44 mission shuttle indicated a molecular film deposit greater than 1 µg/cm² on their -30°C surfaces. It was speculated that similar amounts of contamination may have been deposited on the DSP spacecraft in the shuttle bay (ref.1). It was also speculated that the contaminant originated from the return flux of the shuttle outgassing, since the payload used acceptable outgassing materials, it was extensively outgassed and carefully cleaned. The shuttle bay was also carefully cleaned before launch.

The outgassing deposit, difficult to remove at a temperature of 80°C from the TQCM and later ascertained to be an organic silicone material, was thought to originate from organic silicone released

from the di-methyl-ethoxy-silane (DMES). This is a proofing agent which is sprayed on the shuttle tiles before launch to prevent water absorption.

The following explores the various sources which could have produced the contamination.

- 1. The silicone outgassing from the tiles needed to produce in the bay a flux sufficient to deposit about $1 \,\mu g/cm^2$ during about 4 hours and 45 minutes. This is the time in orbit during which the above deposit occurred. The tile outgassing had to surround the shuttle and backscatter in the bay.
- 2. An estimate of the payload outgassing which could either impinge directly on the TQCM or after being backscattered by the ambient, return to the bay and deposit on the TQCM and the payload itself.

3. An estimate of a contaminant source located near the IOCM which could produce the deposit after being backscattered by the ambient, or by the source scattering within itself. The source could be either a distributed one or a concentrated one as from a venting plume.

Several contaminant sources are investigated using general kinetic theory principles of molecular flow. These analyses can be beneficial for other investigations where contamination deposits may be found and their origin is sought.

Some of the pertinent experimental data from the flight are reported below together with the assumption used for the analyses.

Experimental Data

The shuttle tiles surface has been estimated for these calculations to be about $2 \times 86m^2$ and the shuttle open bay vent at $86m^2$ (ref.2). The shuttle tiles are

sprayed before launch with about 200lb of DMES (ref.3). This high volatility water proofing agent has a vapor pressure of 230 torr at 20°C. It reacts with the tiles silica to form covalent bonds with the silicone (ref. 4). Its mole mass has been taken to be approximately 400 g/mole for calculations. Tiles sprayed with this material at temperatures in excess of 600°C have shown no mass losses (ref. 5,6) when tested in the laboratory.

The RTV 560 silicone adhesive holding the tiles and employed in the estimated amount of 1200 lb. (ref. 7) has been considered as a source of the deposit. The mole mass and the outgassing activation energy of this silicone have been assumed to be about the same as the outgassing from the tiles.

The payload in the shuttle bay in figure 1 has been estimated for this analysis to weigh in excess of 4000 lb. It had been outgassed, protected against contamination, and built with acceptable outgassing materials.

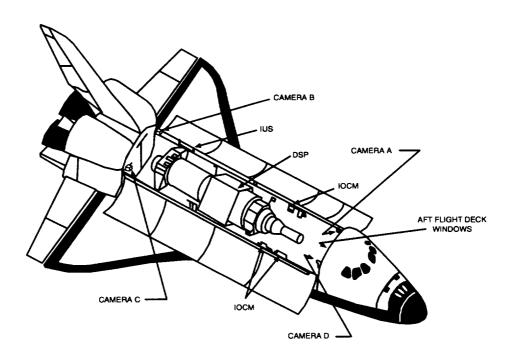


Figure 1. Oblique View of STS-44 Cargo Arrangement

The IOCM package located along the shuttle wall weighed 138kg including the power conditioning unit. It included calorimeters and 6 temperature controlled TQCMs. One of the TQCM had its exposed crystal covered with DC-705 diffusion pump oil to entrap and measure particles in the environment. The vapor pressure of this oil is about 10-8 torr at 20°C producing a calculated evaporating flux of about 6 x 10⁻¹⁰ g/cm²s. The package containing the monitors, with the tape recorders mounted on the adaptive payload carrier had the dimensions of 36"x10"x22", providing a maximum power of 132W. The TOCM #5 facing out of the bay indicated the end-to-end accumulation shown in figure 2 while at a controlled temperature of -30°C to -50°C. The plot suggests an exposure to a high flux of condensible materials. Figure 3a and 3b show the frequency changes during the various events of the flight. As reported, no contamination deposits occurred during the launch and ascent phase. A deposit of .23 µg/cm² occurred after the bay door opening until Attitude Match Update (AMU) maneuver. During the maneuvers and the subsequent deployment of the payload, the accretion was about 1.09 µg/cm². The source of the deposition appeared to originate from outside the orbiter bay given the nature of the events and because the collecting sensor was not viewing the cargo but was viewing out of the bay (ref. 1). This suggested that thruster products or other species released from the orbiter or from the orbiter bay returned to the payload bay. Additional accretions occurred after deployment. These were at lower rates and attributable to attitude and temperature changes or depletion of the outgassing sources. The sensor #5 was heated to 80°C during this period to attempt the removal of the previous contaminant accumulation but

no material was removed. It was noted that after a collision avoidance maneuver requiring engine firing, the rate of contaminant accumulation was about the same as before the maneuver (ref.1). The cumulative mass accretions on the TQCMs including on the #4 TQCM which was covered with oil, are indicated in Table 1 as a function of the events. It is to be noted that this #4 TQCM was losing mass up to the start of the maneuver and then the accumulation occurred. It accumulated a max deposition of 13.3 µg/cm² which is only second to the 28.1 µg/cm² deposition on TOCM #2, which was facing across the bay. The other important data for the analysis are the orbiter altitude and the standard pressure, and density at that orbit. The orbit altitude was 360km, where the pressure is 2.1 x 10⁻⁸ torr and the density is $2 \times 10^8 \text{ cm}^{-3}$ (ref.8).

The atomic oxygen density at that orbit is about $10^8 \,\mathrm{cm}^{-3}$ and the corresponding flux on a surface perpendicular to the orbiter velocity vector will be about 8×10^{13} atoms/cm²/s.

The calculations which follow have been based on a total accumulation of $1 \mu g/cm^2$ occurring at a steady rate during the period going from the bay door opening at 1:32 after launch to 6:18 after launch when the payload was deployed, i.e. for a total time of 4:46 min. (17160 sec.). It is therefore assumed that a constant rate of $1 \mu g/cm^2/17160s = 5.8 \times 10^{-11} g/cm^2/s$ was being deposited on the TQCM.

Return Fluxes Contaminants

1. Shuttle Tiles Outgassing

The pressure of the returning contaminant on the TQCM can be estimated to be 8.55×10^{-10} torr

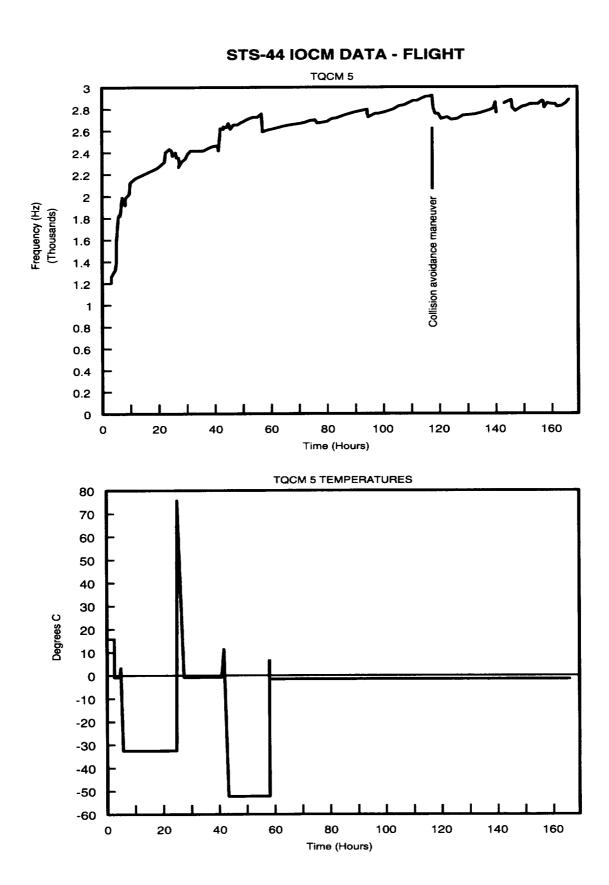
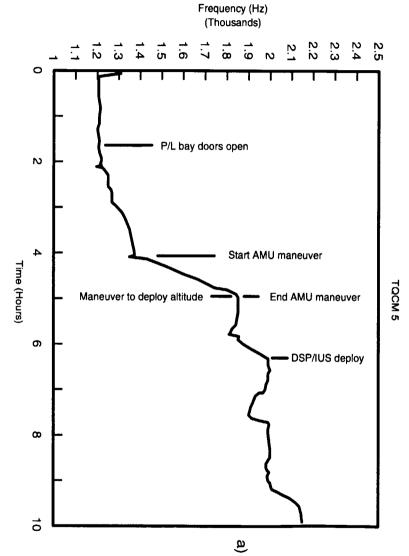
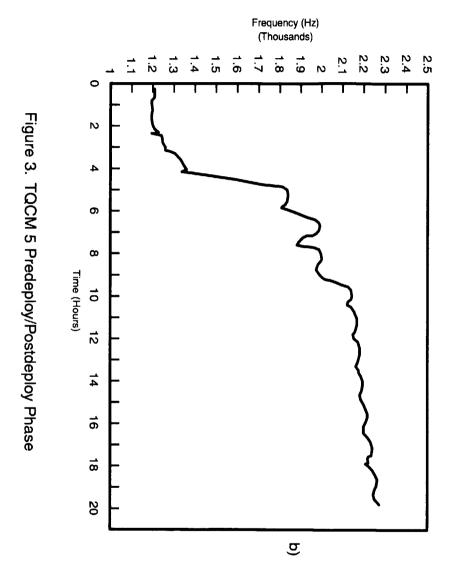


Figure 2. TQCM 5 Flight Data Overview

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STS-44 IOCM DATA - FLIGHT





		TQCM #					
Events	Actual Time	1	2	3	4	5	6
Launch 328/23:44 GMT	00:00	0.00	0.00	0.00	0.00	0.00	0.00
P/L Bay Doors Open	01:32	0.00	0.00	0.05	(0.245)	0.00	0.00
Start AMU Manuever/							
Data Takes	04:00	0.12	0.62	0.25	(0.245)	0.23	0.09
Maneuver to Deploy							
Altitude	04:44	0.62	0.87	(0.03)	0.51	1.01	1.21
IUS/DSP Deploy	06:18	0.90	1.56	0.05	1.04	1.09	1.51
Maximum Deposition	N/A	3.9	28.1	0.16	13.3	3.12	6.24

Table 1. TQCM Cumulative Mass (µg/cm²) Summary: STS-44/IOCM

using the Langmuir relation

$$P_{s} = \phi_{s} (17.14) / \sqrt{\frac{M}{\tau}},$$

with the contaminant flux of $\phi_s = 5.8 \times 10^{-11} \text{ g/cm}^2/\text{s}$, M~400g/mole and T=293°k. If the sticking coefficient of the contaminant on the TQCM was less than 1, then the pressure could have been higher than indicated. The corresponding molecular density at the calculated pressure would be $n_s = 2.68 \times 10^{19} \text{P/}760 = 3.04 \times 10^7 \text{cm}^{-3}$. This pressure and density were the result of reflected piled-up molecules impinging and being accommodated on the TQCM and on other surfaces of the bay. The density of these molecules at a distance ahead of the surface was lower than at the surface and can be estimated to be about 1/64 of

the density at the surface using the relation $n = n_s/4 V_{ac}/V_{in}$ where $V_{ac} = .5$ km/s is the velocity of the molecules accommodated to the surface tem-

perature and $V_{in} \approx 8$ Km/s is the impact orbit velocity. Under these assumptions, the arriving density is $n_r = 4.75 \times 10^5$ cm⁻³, the flux $\phi = nv = 3.8 \times 10^{11}$ cm⁻² s⁻¹ and in terms of mass flux $\dot{m} = \phi M/A = 2.5 \times 10^{-10}$ g/cm²s where M = 400 g/mole and the Avogadro number $A = 6.03 \times 10^{23}$ molec/mole have been used. The above mass flux is the return flux scattered by the ambient density at the 360km orbit. The return flux out of the total outgassing ϕ_D can be estimated using the relation in (ref.9)

$$\phi = \phi_D (V_s/V_D + 1) R/\lambda.$$

This equation for a normal outgassing molecular velocity V_D , orbit velocity V_s , outgass surface radius R, and the mean free path λ for the above orbit indicates a very conservative $\phi_r/\phi_D = 8 \times 10^{-3}$ and $n_r/n_D = 6 \times 10^{-4}$. The density of the molecules before scattering would then be about $n_D = n_r/6 \times 10^{-4}$.

 $10^{-4} = 7.9 \times 10^{8}$, corresponding to $P_D \sim 2.2 \times 10^{-8}$ torr. The flux would be $\phi_D = n_D V/4 \sim 9.87 x$ $10^{12} \,\mathrm{cm}^{-2} \,\mathrm{s}^{-1}$ and the mass rate $\,\dot{m}_D = 6 \,\mathrm{x} \, 10^{-9} \,\mathrm{g/cm}^2 \mathrm{s}$. These should be the parameters of the molecules surrounding the shuttle which after being scattered by the ambient density and piled up in the bay would deposit on the TQCM and on other surfaces in the bay. A representation of the calculated parameters at the respective locations is shown in figure 4. It shows that, for the tiles or any other source associated with the tiles, to be the sources of the return flux, they had to be outgassing at a rate of 6 x 10⁻⁹ g/cm²/s, producing a density on their surface of about 7.9 x 108 cm⁻³ and a pressure of about 2.2 x 10⁻⁸ torr. These conditions should be present above the bay as a result of the shuttle rotating and producing in this manner a cloud of those magnitudes around the shuttle.

The pressure in the bay resulting from this return flux and the ambient pressure at 360km, can be calculated to be about 1.4×10^{-6} torr. It is obtained by adding the accommodated ambient pressure previously indicated, $P = 64 \times 2.1 \times 10^{-8} = 1.38 \times 10^{-6}$ torr and the return flux pressure, 8.5×10^{-10} torr. These pressures compare to the measured in-flight bay maximum pressure of 4.4×10^{-4} and minimum of 8.3×10^{-6} torr.

An estimate of the tile outgassing based on the general acceptance of materials for space application can be carried out in the following manner. The intent is to compare the rate obtained to the rate calculated to be needed for the return flux, $\dot{m} = 6 \times 10^{-9} \text{ g/cm}^2/\text{s}$. The 90 x 10³g (200lb) of DMES sprayed on the tile surface of about A = 172 x 10^4 cm^2 , indicates a DMES deposit per unit area

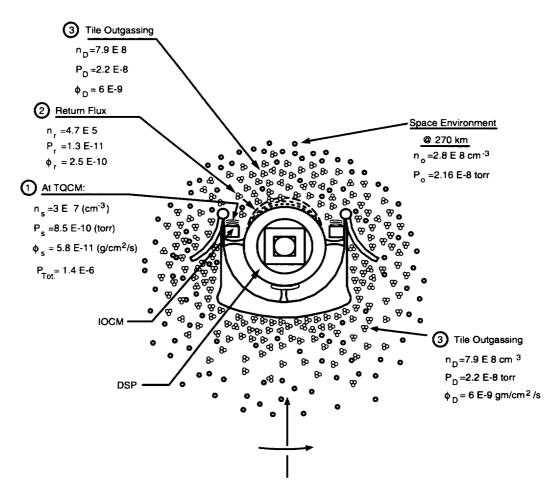


Figure 4. Tile Outgassing Return Flux

of 5.23 x 10⁻² g/cm². The vapor pressure of the DMES at 20°C is 230 torr (ref.3). Accordingly, the rate of evaporation using Langmuirs' equation with a mole mass of 400g/mole, at 20°C will be 15.58 g/cm²/s and the DMES would evaporate almost immediately. Consequently, any outgassing from the tiles must originate from a reacted product of the tile silica and the DMES. The reaction products must have a very strong bonding, since tests of sprayed tiles showed no mass loss at 600°C and beyond as shown by the Thermo-gravimetric Analysis (TGA) thermogram (figure 5). Assuming that one-third of the 90kg DMES reacted with the tile and the resulting compound meets the ASTM E595 criteria of $m/m_0 = 0.1\%$ for the volatile condensable material on a surface at 25°C in vacuum, the outgassing rate can be obtained with the assumption

that the outgassing activation energy is about E = 20 kcal/mole. This relatively high energy is justified by the inability to remove the deposit from the TQCM at 80°C temperature. It does not cover additional changes in energy resulting from the material exposure to UV and/or atomic oxygen. So, the tiles outgassing rate m_0 can diffuse out of the tile according to (ref.10)

$$\dot{\mathbf{m}} = \mathbf{km_0} e^{-\mathbf{E}/\mathbf{R}T} / \sqrt{t} \ (\mathbf{g}/\mathbf{hr})$$

where k is the constant to be determined, E = kcal/mole is the activation energy, R = 1.98 cal/ °K mole, the gas constant, T = 398 °K is the outgassing temperature, t = 24 hrs = 1 day the time and m_0 is the initial mass of the material being outgassed. Using the parameters for the ASTM 595 test, the integration of the above from

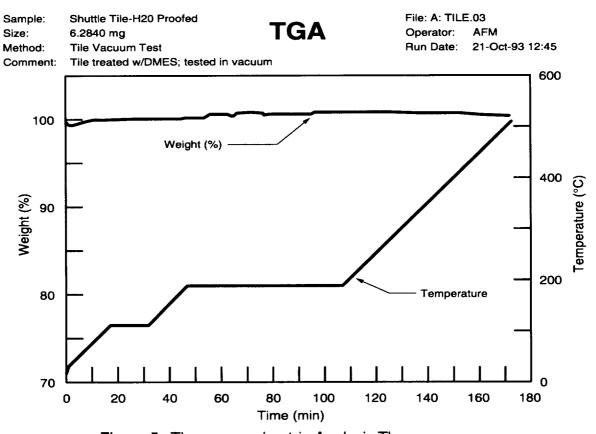


Figure 5. Thermo-gravimetric Analysis Thermogram

t = o to t provides the constant

$$k = 1/2 \text{ m/m}_0 (e^{-E/RT} t^{1/2})^{-1} = 5.98 \text{ x } 10^7 t^{-1/2}$$

where t = 1,2,3.. days. The outgassing rate at 293K for $m_o = 1/3(90)$ kg is then $\dot{m} = 1.84 \times 10^{-8} / \sqrt{t}$ (g/s) or in pv units $Q = \dot{m} P_o V_o / M = 7.83 \times 10^{-7} / \sqrt{t}$ (torr //s). The outgassing rate per unit area of tile is $\dot{m} / A = 1.06 \times 10^{-14} / \sqrt{t}$ (g/cm² s) = 4.55 x $10^{-13} / \sqrt{t}$ (torr //cm²s).

This outgassing rate $1.06 \times 10^{-14}/\sqrt{t}$ g/cm²/s is much less than the required $\dot{m} = 6 \times 10^{-9}$ g/cm²/s for the return flux. Accounting for the various assumptions with regard to energy, temperature, fraction of condensable, and the amount of outgassing material may change the results. However, it appears improbable that those changes could account for the four orders of magnitude between the two rates. From these results, the return flux of tile outgassing is not the origin of the contamination.

A similar calculation of the outgassing of the RTV-560 silicone adhesive, indicates an outgassing rate per unit area of $3.25 \text{ E}-13/\sqrt{t}$ when a total coverage area of the tiles (172m^2) is assumed. The calculation is based on total outgassing of 1/3 the 1200 lb. (560 kg.) of RTV and a Collected Volatile Condesable Material (CVCM) as per the ASTM-E-595 test (ref. 11) of 0.5% on a 25°C surface. This rate is also much smaller than the 6E-9 g/cm²/s required for the deposit to originate from a return flux.

2. Payload Outgassing

Assuming that the payload is the source of the return flux, one can calculate its outgassing using the method used for the tile outgassing. For a payload of 600kg and an outgass source of m = 1/3 (600) kg with the same assumptions as before, the outgassing @ 20° C is $\dot{m}_{s/c} = 3.18 \times 10^{-2}$ /

 \sqrt{t} g/day = 3.68 x $10^{-7}/\sqrt{t}$ g/s = 1.56 x $10^{-5}/\sqrt{t}$ torr //s. The flux out of the bay door opening, assumed to be 82 m², will be $\phi_D = 3.68 \times 10^{-7}/8.2 \times 10^5 = 4.48 \times 10^{-13}/\sqrt{t}$ g/cm²/s. The return flux fraction as before is $\phi_r = 8E - 3$ $\phi_D = 3.6$ E- $15/\sqrt{t}$ g/cm²/s. This is considerably less than the required depositing flux of 5.8 x 10^{-11} g/cm²/s at the TQCM and the return from the payload outgassing should also be excluded as the source of contaminant.

The pressure in the bay due to the payload return flux can be estimated by the following:

The conductance of the bay opening (86m^2) taken as to be that of an orifice is $C = 11.6 \sqrt{28/400}$ x $86 \times 10^4 = 2.26 \times 10^6$ //s. Then the bay pressure is $P = \dot{m}_{s/c}/C = 6.9\text{E}-12/\sqrt{t}$ (torr) and the density is $2.4\text{E}5(\text{cm}^{-3})$. Again, this pressure is lower than the 8.55 E-10 estimated to be required for the contaminant deposition.

3. Other Outgassing Sources

A return from a surface outgassing at a rate of 6 x 10^{-9} g/cm²/s = 2.55 x 10^{-7} torr //cm²/s near the TQCM would provide the needed return. This rate of outgassing may be expected from the initial outgassing of materials, such as silicone rubbers, polyamides, and epoxies.

One may consider a return from a concentrated venting plume exiting from an opening near the TQCM location. In fact, if one assumes a 1 cm² vent from a box, with an outgassing flow of 6 x 10^{-9} g/s, the scattered return could amount to a local flux of 5.8×10^{-11} g/cm²/s depositing on the TQCM. One may calculate that the pressure in the box producing the above vent flow could be about P ~ 8.4×10^{-8} torr. This pressure can be easily maintained by several cm² of outgassing

polymers in a box with a 1 cm² vent opening. The same outgassing flow could exit from several leak openings of the box and produce the expected return. In fact, we may estimate, as previously done, the outgassing from the 138kg IOCM package with the indicated dimensions. When this is done, the outgassing at 20°C would be $2.8E-8/\sqrt{t}$ g/s = $(1.19 \times 10^{-6}/\sqrt{t})$ torr t/s. This flow rate issuing from a 1 cm² vent leak C = 3.04(t/s) would be maintained by a pressure of $3.9 \times 10^{-7}/\sqrt{t}$ torr which again is a reasonable pressure to exist in the box for a period of time.

Contamination from Other Than Return Fluxes

The contamination deposit on the TQCM could have been produced by a direct flux from a nearby surface or from a flux reflected from a nearby surface. In the absence of details of the payload components about outgassing sources and view factors between them and the TQCM one cannot speculate on those possible sources. To note, however, that the report was specific in pointing out the clean conditions of the payload and shuttle bay. These sources, however, should be considered: 1) an outgassing source originating from below or around the package which would self scatter above the TQCM or 2) the scatter within a vent plume exiting from the package through a narrow passage.

A self-scattered flux must produce on the TQCM a flux of 5.8×10^{-11} g/cm²/s which is equivalent to about 8.73×10^{10} cm⁻² s⁻¹. The source to produce this scatter magnitude can be roughly estimated by using the relation developed by the author (ref.12)

$$\phi_D = ((\phi_{ss}^2/1.78 \times 10^{-2} (\sigma R/V_D))^{1/2} cm^{-2} s^{-1}$$

where $\phi_{ss} = 8.73 \times 10^{10}$, $\sigma \approx 10^{-15} \text{ cm}^2$ is the cross section of the molecules $V_D \sim 4 \times 10^4$ cm/s is the molecule velocities and R ~ 50cm is the approximate radius dimensions of the outgassing surface. The relation indicates that a flux of about $(2 \times 10^{15} \text{ cm}^{-2} \text{ s})$ or about 1.32 x $10^{-6} \text{ g/cm}^2/\text{s}$ is required. This is a large outgassing rate and an improbable source of the deposit, unless there is a large source very near the TQCM. Such a source could be produced from a leak or vent opening. In fact, again assuming for calculations the existence of a 1 cm² leak orifice in a container, the above source leak would be produced by an outgassing pressure of 1.83 x 10⁻⁵ torr. This is a possible pressure in a semi-closed volume with many internal outgassing surfaces.

The other possibility is the scattering within a plume at a location just above the TQCM. Assuming the origin of a plume venting at a rate of $\dot{m} = 1.3 \times 10^{-6} \text{ g/cm}^2/\text{s}$, located at a distance of 10cm from the TQCM, the flux 1cm above the TQCM, i.e., at an angle $\theta = 84^{\circ}$ can be estimated (ref.13) for n = 1 to be

$$\phi = \dot{m} (n+1/2\pi r^2) \cos^n \theta = 4 \times 10^{-10} \text{ g/cm}^2/\text{s}.$$

If one further assumes that of the above flux 1/4 to 1/6 is directed toward the TQCM, the imping flux can be 1 x 10^{-10} > ϕ > 6.7 x 10^{-11} which is in the range of the needed flux at the TQCM.

Conclusions

A survey of the possible sources of contamination and their transports to the shuttle bay surfaces have been investigated. The contaminants were detected and monitored by a TQCM at -30°C pointed out of the bay. The deposit of about 1 µg/cm² occurred

during the early hours of the flight after the bay doors opening and before the release of a payload. The deposits were attributed to the ambient scattered return flux of the outgassing from the shuttle tiles. The shuttle tiles were sprayed before launch with a H₂O repellent product (DMES), and the payload and bay had been carefully monitored for cleanliness.

The analyses carried here have indicated the following.

- •The tiles outgassing had to be about 6 x 10⁻⁹ g/cm²/s for it to be scattered at the 360km orbit, returned to the bay and deposited at the recorded rate. Outgassing tests of the sprayed tiles indicated extremely small mass loss at temperature in excess of 600°C. Also, a theoretical estimate of the outgassing based on material acceptance criteria for space application, indicated rates of the order of 10⁻¹⁴ g/cm²/s much lower than those needed for the return and indicated deposits.
- •The outgassing of the 1200 lb. (560 kg) of the RTV-560 tile adhesive was calculated at 10⁻¹³ g/cm²/s when conservatively assumed to be completely exposed. This rate is about four orders of magnitude lower than the required source from the return flux.
- •The payload outgassing return flux was considered and calculated to be of the order of 10⁻¹⁵ g/cm²/s less than the indicated outgassing deposit rate at the TQCM of about 10⁻¹¹ g/cm²/s.
- •Return fluxes from sources located near the monitor package, below, above, or adjacent with outgassing rate of the needed magnitude were investigated. Sources from a small vent of a volume containing a variety of materials could produce the deposit. The pressure in the volume

to maintain the needed outgassing rate would be about 10⁻⁷ torr. This is a pressure to be expected in a volume with a limited venting conductance and several outgassing materials.

•Other transports of outgassing products to TQCM from the self-scattering of distributed outgassing sources or from the scatter within a vent plume have been investigated for possible contaminant sources.

It has been shown that there are several possible sources and transports for the contaminant deposits on the TQCM. All those sources must be somewhere near, above, or below the monitor package. The tiles and the tile holding RTV outgassing and their return fluxes into the bay do not appear to be the sources of the deposits. In the absence of measurements at other bay locations, it is impossible to characterize the source, the mechanism, and the location for the contamination deposit.

Additional on-orbit measurements at various orbit altitudes, for different bay orientation and solar energy input, are needed.

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13. ABSTRACT (Maximum 200 words)		

The sources of molecular deposits on a Temperature-Controlled Quartz Crystal Microbalance (TQCM) attached to the shuttle bay wall at some distance from the payload and pointing out of the bay have been investigated. Explored as possible sources are: (1) the outgassing of the shuttle tiles treated with the water proofing di-methyl-ethoxy-silane (DMES) compound, scattered back by the ambient molecules into the bay; (2) The ambient scattered return flux of the outgassing from the RTV 560 silicone which is used as the adhesive for the tiles; (3) the ambient scattered return flux of the payload outgassing; (4) the return flux from outgassing source originating near, below, or adjacent to the package containing the TQCM; and (5) the self-scattering of venting plumes and outgassing above the monitor package depositing on the TQCM. In all of these sources, the magnitudes of the outgassing, of the leakage rates, and venting had to be estimated in the absence of data. The resulting analyses which can be useful for similar investigations, indicates that the outgassing from the tiles, the RTV, the payloads, or the shuttle may not have been the sources of the deposit (about 1 mg/cm² during the period from bay door opening and payload release from the bay). It appears more probable that the origin is the self- and/or ambient-scattered return fluxes originating near, below, or internal to the monitor package.

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